IFW

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of

BEGUIN et al.

Atty. Ref.: 1721-84

Serial No. 10/523,397

Group: 2878

Filed: February 3, 2005

Examiner: Unassigned

For: METHOD FOR OPENING CARBON NANOTUBES AT THE ENDS THEREOF AND IMPLEMENTATION

* * * * * * * * * * * *

July 26, 2005

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

SUBMISSION

Submitted herewith is a copy of the International Preliminary Examination Report in the English language for the above-referenced application.

Respectfully submitted,

NIXON & VANDERHYE P.C.

By: _____

B. J. Sadoff
Reg. No. 36,663

BJS:pp
901 North Glebe Road, 11th Floor
Arlington, VA 22203-1808
Telephone: (703) 816-4000
Facsimile: (703) 816-4100

Translation

PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference CP60754	FOR FURTHER ACTION	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
International application No. PCT/FR2003/002499	International filing date (*day/month/year*) 08 août 2003 (08.08.2003)	Priority date (*day/month/year*) 08 août 2002 (08.08.2002)
International Patent Classification (IPC) or national classification and IPC C01B 31/02		
Applicant CENTRE NATIONAL DE LA RECHERCHE SCIENTIFIQUE (C.N.R.S.)		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 7 sheets, including this cover sheet.

☐ This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of _____ sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☐ Certain observations on the international application

Date of submission of the demand 08 mars 2004 (08.03.2004)	Date of completion of this report 18 February 2005 (18.02.2005)
Name and mailing address of the IPEA/EP Facsimile No.	Authorized officer Telephone No.

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.
PCT/FR2003/002499

I. Basis of the report

1. With regard to the **elements** of the international application:*

☒ the international application as originally filed

☒ the description:

pages 1-8, as originally filed

pages _____, filed with the demand

pages _____, filed with the letter of _____

☒ the claims:

pages 1-9, as originally filed

pages _____, as amended (together with any statement under Article 19

pages _____, filed with the demand

pages _____, filed with the letter of _____

☒ the drawings:

pages 1/4-4/4, as originally filed

pages _____, filed with the demand

pages _____, filed with the letter of _____

☐ the sequence listing part of the description:

pages _____, as originally filed

pages _____, filed with the demand

pages _____, filed with the letter of _____

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.
These elements were available or furnished to this Authority in the following language _____ which is:

☐ the language of a translation furnished for the purposes of international search (under Rule 23.1(b)).

☐ the language of publication of the international application (under Rule 48.3(b)).

☐ the language of the translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

☐ contained in the international application in written form.

☐ filed together with the international application in computer readable form.

☐ furnished subsequently to this Authority in written form.

☐ furnished subsequently to this Authority in computer readable form.

☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. ☐ The amendments have resulted in the cancellation of:

☐ the description, pages _____

☐ the claims, Nos. _____

☐ the drawings, sheets/fig _____

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).**

** Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rule 70.16 and 70.17).*

*** Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.*

V. **Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

1. Statement

Novelty (N)	Claims	2-5, 7, 8	YES
	Claims	1, 6, 9	NO
Inventive step (IS)	Claims		YES
	Claims	1-9	NO
Industrial applicability (IA)	Claims	1-9	YES
	Claims		NO

2. Citations and explanations

Reference is made to the following documents:

D1: SMITH M. R., BITTNER E., BOCKRATH B: "Hydrogen storage on carbon single-walled nanotubes" CARBON'01, PROCEEDINGS OF THE 25TH INTERNATIONAL CONFERENCE ON CARBON, PAPER NO. 36.5, LEXINGTON, KY: AMERICAN CARBON SOCIETY, 12 July 2001 (2001-07-12) 18 July 2001 (2001-07-18) XP008026875.

D2: DILLON A D ET AL: "A SIMPLE AND COMPLETE PURIFICATION OF SINGLE-WALLED CARBON NANOTUBE MATERIALS" ADVANCED MATERIALS, VCH VERLAGSGESELLSCHAFT, WEINHEIM, DE, vol. 11, no. 16, 10 November 1999 (1999-11-10) pages 1354-1358, XP000875153 ISSN: 0935-9648.

I - Observation:

Document D1 contains two pages that have not been numbered. For the purpose of clarity, the pages of D1 have been numbered as page 1 to page 4.

II - Novelty

2.1 Claims 1, 6 and 9:

Document D1 describes a comparative study on the hydrogen adsorption capacity of single-walled carbon nanotubes, which study was carried out on two batches of commercial carbon nanotubes (D1, page 1, right-hand column, last paragraph):

- a first batch, which is referred to as the "Prepared Grade" batch and can be described as a raw material; and
- a second batch, which is referred to as the "Purified Grade" batch and has already been exposed to an oxidation step with 2.6 M nitric acid (15% by weight) (see D1, page 3, left-hand column).

A nitric acid oxidation step followed by a gas-phase oxidation step:

D1 considers the adsorption of hydrogen onto said second batch *per se* then onto the same batch but which has, in addition, been exposed to a second oxidation step with CO2 flow at approximately 600°C (D1, pages 3 and 4). The duration of this second oxidation step is not specified.

D1 emphasises that this second oxidation step leads to a significant increase in the hydrogen storage capacity of the second batch that has been treated with CO2 flow (D1, page 4, right-hand column, last paragraph and page 4, figures 6 and 7), in

comparison with the same batch that has not been treated with said CO2 flow.

Figure 6 and 7 (D1, page 4, right-hand column) illustrate the increase in the storage capacity of carbon nanotubes that have been exposed to:

- a first liquid-phase oxidation step (2.6 M nitric acid; 12 hours); and
- a second gas-phase oxidation step (CO2; 600°C).

Concentrated acid:

The expression "concentrated acid" has a relative meaning that does not enable the subject matter in claim 1 of the present application to be delimited with respect to D1 (the PCT International Search and Preliminary Examination Guidelines, page 44, paragraph 5.34).

Open carbon nanotubes:

It is not explicitly disclosed that the ends of nanotubes in the "Purified Grade" batch are open.

However, a person skilled in the art is fully aware that synthesis methods produce closed carbon nanotubes and that said nanotubes can be opened chemically via, in particular, a liquid-phase acid oxidation step.

In D1, when the H2 adsorption of the "Purified Grade" batch *per se* (curve A, fig 5) is compared with that of the same batch treated with helium flow

at 700°C, it is stated that (page 3, right-hand column) the slight difference in adsorption in favour of the temperature-treated material can be reasonably ascribed to the removal of the functional groups that block the adsorption sites. However, D1 makes it clear that, in view of the mean diameter of said nanotubes (1.2 nm), it is highly unlikely that these groups could have blocked the nanotube openings. This means that said nanotubes are open. The ends of said nanotubes are removed by the oxidation treatment to which the "Purified Grade" batch is exposed.

In short, D1 describes a study concerning the adsorption of carbon nanotubes that have been exposed to a liquid-phase oxidation step carried out in an acid (and leading to the opening of said nanotubes) followed by a gas-phase oxidation step. This two-step oxidation process opens and purifies the carbon nanotubes.

As a result, the subject matter of claims 1, 6 and 9 is not novel.

3 - **Inventive step:**

3.1 Claims 2, 3 and 5:

Document D1, which is considered to be the closest prior art, therefore describes a method in accordance with the present claim 1 but used with single-walled carbon nanotubes.

In light of document D1, it would be obvious for a person skilled in the art to apply this method, with

a corresponding effect, to multi-walled nanotubes.

It follows that the subject matter of claims 2, 3 and 5 is not considered to be inventive.

3.2 Claim 7:

Document D1 mentions an oxidation step with CO2 flow at approximately 600°C but does not specify the duration thereof. The only parameter concerning said duration, as indicated in the present claim 7, cannot, however, render said claim inventive because a fundamental portion of its subject matter, i.e. the use of CO2 between 500°C and 600°C, is already known from D1.

The selection of this duration would appear to be a standard step to a person skilled in the art. What is more, no unexpected effects or properties resulting from the selection of said duration are indicated in the application.

The subject matter of claim 7 is not, therefore, considered to be inventive.

3.3 Claim 4:

The selection of an acid having a concentration of 60-75 % by weight would appear to correspond more to a selection intended to shorten the duration of the reflux oxidation step, i.e. a duration of 30 to 50 minutes in the present application as opposed to 12 hours in document D1, than to a selection that leads to a special technical effect.

As a result, the subject matter of the above claim is not considered to be inventive.

3.4 Claim 8:

Document D1 does not disclose an intermediate step of filtering and washing the nanotubes, which were exposed to the first liquid-phase oxidation step.

However, filtering reflux-oxidised carbon nanotubes and washing same with distilled water is a conventional step, which is not only normal in oxidation treatments of this kind but also well known to a person skilled in the art. Document D2, for example, discloses such an intermediate step (see page 1354, right-hand column, last paragraph) carried out between a first step involving the reflux oxidation of the nanotubes in a nitric acid solution, and a second step of oxidation in air.

It follows that the subject matter of claim 8 is not considered to be inventive.